Liquid penetration of freeze-drying and air-drying wood of plantation Chinese fir

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Abstract: A comparative study was conducted on liquid penetration of the freeze-drying and air-drying sapwood and heartwood lumber of plantation Chinese fir (*Cunninghamia lanceolata*). The maximum amount of dyeing solution uptake by the capillary rise method was used to evaluate the liquid penetration properties of the treated wood. The pit aspiration ratio was determined by semithin section method. Changes in wood microstructure were investigated using scanning electron microscopy. The results showed that compared with air drying, the freeze drying had a significant effect on liquid penetration of sapwood and heartwood of Chinese fir. The liquid penetration of sapwood is significantly higher than that of the heartwood for both drying treatments. Low pit aspiration ratio and cracks of pits membrane of some bordered pits are the main reasons for increasing liquid penetration after freeze drying treatment.

Keywords: Chinese fir; Sapwood lumber; Heartwood lumber; Freeze drying; Air drying; Liquid penetration; Pit aspiration ratio
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Introduction

Chinese fir ((*Cunninghamia lanceolata* (Lamb.) Hook.) is a major plantation species in China. Currently the total planting area of Chinese fir in China is approximately 5.2 million hm^2. However, due to the rapid growth rate, short rotation and the large juvenile wood proportion, the wood quality of plantation grown Chinese fir is much lower than that of the natural, particularly in the aspects of wood density and surface hardness. Therefore, to utilize the plantation Chinese fir more efficiently, various modifications are needed to improve its density, strength and surface hardness. On the other hand, the plantation Chinese fir is really a refractory species. Usually it requires very high pressure to make the chemicals or resins impregnate into the wood, while high pressure could result in damage and deformation of the wood. It has been reported that the liquid permeability varied depending on drying methods and pre- or post- treatments which could improve it (Matsumura *et al.* 1995a, 1995b, 1996; Zhao *et al.* 2003), but few papers were reported on the effect of different drying methods on liquid penetration of wood. Therefore, understanding the mechanism of drying methods on liquid penetration of plantation Chinese fir is the key project to make the high quality solid wood products through various chemical impregnation treatments.

The basic principle of freeze drying is that a substance can evaporate directly from the solid state without passing through a liquid state. In freeze drying, water is converted to ice by freezing, subsequently, ice is converted directly to vapour. This type of evaporation, characteristic of freeze drying, is called sublimation. Because freeze drying method can preserve the nutrition components and biological properties of dried materials to the most extent, it is widely used in many industries (Xu *et al.* 1994; Ye *et al.* 2000; Wang *et al.* 2002; Li *et al.* 2004), such as, pharmaceutical manufacturers, food processors, taxidermy, and the floral industry. However, since during freeze drying process, the velocity of mass and heat transfer is very low, the consumed energy is high and the drying time is long, the research of using freeze drying method to dry wood is limited. Although currently the freeze drying is impractical on commercial scale in wood drying industries, it is still an important experiment means to clarify the fluid flow mechanism in wood.

Materials

Fifteen Chinese fir trees were cut in Jiangxi Province. Under the same planting condition, five trees were selected for fast growth speed (breast diameter more than 25 cm), medium growth speed (23–25 cm breast diameter range), low growth speed (breast diameter less than 23 cm), respectively.

Methods

Specimen preparation

Logs below 1.3 m height were cut from each tree. South part of logs was used for drying experiments. Two to three pieces of heartwood and sapwood boards with a length of 1.3 m and a thickness of 30 mm were prepared by sawing from pith to bark. Then these boards were cut into lumbers with a width of 80 mm and a thickness of 25 mm. Finally, along the tree growth direction, the lumbers were cut into sticks with a length of 20 cm and the sticks were divided into A and B part with proportion of 2:1 in the width direction. Fifteen A-part sticks (with dimension of 200 mm × 50 mm× 25 mm) and 15 B-part sticks (with dimension of 200 mm × 25 mm× 25 mm), , cut separately from heartwood and sapwood, were treated by air drying and freeze drying, respectively.

Drying treatments

Vacuum freeze drying machine (FD-1) was used for freeze drying. The condensation temperature is –52°C and the vacuum degree is 5Pa. The final moisture content of heartwood and sapwood specimens after freeze drying treatment is 12.83% and

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11.35% respectively. For the air drying, boards were exposed to well-ventilated environment for 97 days. The final moisture content of heartwood and sapwood specimens after air drying treatment is 10.07% and 8.51% respectively.

Measurement of penetration

Specimens (100 mm × 25 mm × 15 mm) for liquid penetration experiment were cut from the middle part of each drying sample. During cutting process, the knot, surface checks and interior checks should be avoided to reduce the effect of specimen defects on liquid penetration. All the specimens were stored in the condition room (20 ℃, 65%) for reaching the equilibrium of moisture content. Before the measurement, the tangential and radial surfaces of the specimens were coated with the epoxy resin. Specimens were placed in a standing position on one side of the cross section at the surface of dyeing solution. The dyeing solution used in the experiment is patent blue (C.I.: 42045) with 0.2% concentration. By weighing the specimens every period of time, the amount of penetration was denoted by the difference of weight. The amount of penetration in specimens was determined by the capillary rising method for 512 min, then the time (min) and liquid uptake ($g \cdot cm^{-2}$) relation curves were presented.

Determining the pit aspiration ratio

Optical microscopy was used to determine the ratio of pit aspiration. Semithin section samples of earlywood and latewood were cut from heartwood and sapwood after drying treatments. For each observation sample, pit aspiration ratio was calculated from 50 pits.

SEM observation

Scanning electron microscopy (SEM) was used to observe the tracheids wall and bordered pits characteristics in freeze drying and air drying wood. Sample of the radial-longitudinal surface were prepared by splitting, mounting, and coating them with gold prior to observation.

Results and discussion

liquid penetration of freeze drying and air drying wood

Figure 1 and 2 showed the solution uptake by sapwood and heartwood of plantation Chinese fir after treated by freeze drying and air drying. It showed that the solution uptake of freeze drying samples was obviously higher than that of the air drying samples. The wood samples showed a marked increase in uptake with longer times. During the early period of penetration process, the speed of solution uptake was high, but the uptake speed decreased with increase of penetration time. The maximum solution uptake value of the sapwood was 1.78 $g \cdot cm^{-2}$ for freeze-dry samples and 1.13 $g \cdot cm^{-2}$ for air-dry samples (Fig. 1), while that of the heartwood was 0.27 $g \cdot cm^{-2}$ for freeze-dry samples and 0.16 $g \cdot cm^{-2}$ for air-dry samples (Fig. 2). The variance of analysis showed that the effect of freeze drying and air drying on liquid penetration of sapwood and heartwood of plantation Chinese fir is very significant (Table 1). For both freeze drying and air drying method, there are very significant difference in amount of liquid uptake between the sapwood and heartwood of Chinese fir (Table 2).

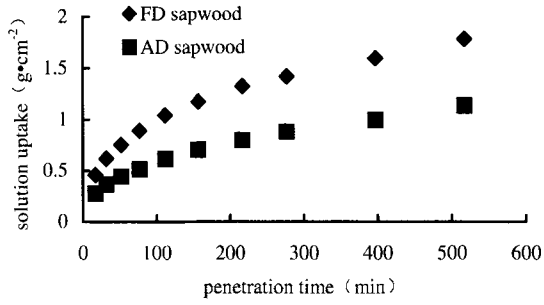

Fig. 1 Solution uptake in Chinese fir sapwood specimens treated by freeze-drying and air-drying methods

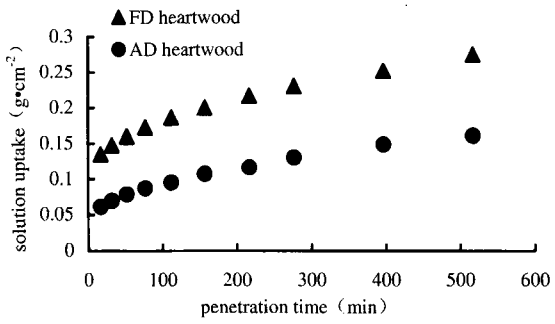

Fig. 2 Solution uptake in China-fir heartwood specimens treated by freeze-drying and air-drying methods

Table 1. Variance of analysis for liquid uptake between freeze drying and air drying

Source	df	SS	MS	F	S
Block	14 (14)	1.6069 (0.0430)	0.1148 (0.0031)	1.27 (2.38)	
Treatment	1 (1)	3.1403 (0.0976)	3.1403 (0.0976)	34.82 (75.63)	** (**)
Error	14 (14)	1.2627 (0.0181)	0.0902 (0.0013)		
Total	29 (29)	6.0099 (0.1587)			

** 0.01 level significant. Out of bracket is sapwood value, in bracket is heartwood value.

Table 2. Variance of analysis for liquid uptake between sapwood and heartwood

Source	df	SS	MS	F	S
Block	14 (14)	1.1497 (0.2995)	0.0821 (0.0214)	0.98 (0.97)	
Treatment	1 (1)	17.0559 (7.1303)	17.0559 (7.1303)	203.47 (324.25)	** (**)
Error	14 (14)	1.1736 (0.3079)	0.0838 (0.0220)		
Total	29 (29)	19.3792 (7.7376)			

** 0.01 level significant. Out of bracket is air drying value, in bracket is freeze drying value.

Pit aspiration ratio of freeze drying and air drying wood

Most researches support the theory that the bordered pit is the primary structure governing the permeability of softwood (Comstock 1967; Petty 1970; Kuroda and Siau 1988). Three factors are effective in reducing the capillary size of the pit pairs, and any combination of these may be present in a wood species. These factors are pit aspiration, pit occlusion with extractives,

and pit incrustation (Flynn 1995). Table 3 showed the pit aspiration ratio of earlywood and latewood in sapwood and heartwood treated by freeze drying and air drying methods. During freeze drying, pit aspiration may be avoided or reduced, due to the elimination or reduction of surface tension forces. In freeze drying, the green wood is frozen and evacuated under an absolute pressure of less than 0.46 cmHg which corresponds to the triple point of water. Below the ripple point of water, ice sublimes directly to vapor with no liquid phase. Surface tension forces occurring at the water-air interface where air drying occurs are thereby eliminated (Siau 1984). Our study found that the pit aspiration ratio is higher in the heartwood than sapwood and also higher in the earlywood than latewood. This result accords to the prior finding that the liquid penetration of sapwood is better than those of heartwood (Erickson 1970), and the liquid penetration of latewood is better than those of earlywood (Petty 1970).

Table 3. Pit aspirated ratio of earlywood and latewood in sapwood and heartwood treated by freeze drying and air drying methods

Drying methods	sapwood		heartwood	
	earlywood	latewood	earlywood	latewood
freeze dry	0.44	0.30	0.64	0.48
air dry	0.64	0.52	0.82	0.60

Microstructure observation of freeze drying and air drying wood

Figure 3 and 4 showed the results of the SEM observations of the tracheids wall and pits of freeze drying and air drying wood samples, respectively. In Figure 3, it is evident that cracks were occurred in the pits membrane. This could be considered as amplifying the effect radius of pit membrane pores and therefore increasing permeability. Whereas, in Figure 4 the bordered pits were kept intact, and this may be explained by the fact that air drying is a gentle drying process.

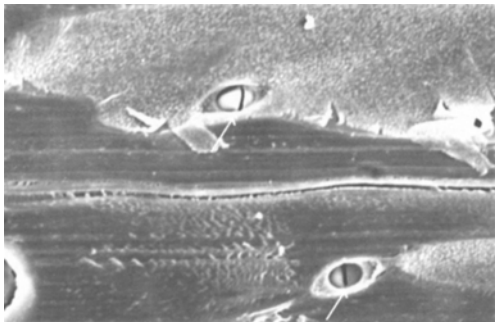

Fig.3 Bordered pits Cracks in sapwood after freeze drying (x 2700)

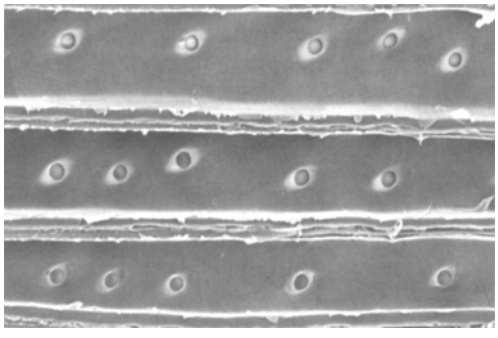

Fig.4 No damage in pits membrane of sapwood after air drying (x 1100)

Conclusions

The variance of analysis showed that the effect of freeze drying on liquid penetration of Chinese fir sapwood and heartwood is very significant. The better liquid penetration of Chinese fir wood after freeze drying could be attributed to the fact that the pit aspiration ratio is lower during freeze drying process. In addition, cracks were found in the pit membrane of sapwood specimens after freeze drying, which could be considered as the other factor contributing to the significant increase of liquid penetration.

The liquid penetration of Chinese fir sapwood after freeze drying and air drying is always higher than those of heartwood. The variance of analysis showed that for both drying methods, there are significant difference in amount of liquid uptake between Chinese fir sapwood and heartwood, which could be attributed to the pit occlusion with extractives and pit incrustation during the heartwood formation besides the lower pit aspiration ratio in dried sapwood.

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